END-OF-YEAR-REPORT

MICROMECHANICAL PROPERTIES OF BERYLLIUM AND OTHER INSTRUMENT MATERIALS

by

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MICROMECHANICAL PROPERTIES OF BERYLLIUM AND OTHER INSTRUMENT MATERIALS

This research program was initiated on October 1, 1977. It is sponsored by the Office of Naval Research. The objective of the program is to evaluate and understand the micromechanical properties of beryllium and other instrument materials for use in gyroscopes, so that dimensional instability can be improved. Improved dimensional stability is expected to lessen the need to periodically align gyroscopes in service. Drift in alignment has been attributed in part to mass shifts of 10⁻⁶ inches in critical components of gyroscopes.

This program has two major thrusts. One is to measure micromechanical properties at strains of less than 10^{-7} . Properties of interest include microyield stress, microcreep rate, and microstrain hardening. The other major thrust is to measure micromechanical properties at strains of less than 10^{-8} .

This program is carried out in cooperation with materials engineers and gyroscope designers on the staff of the C. S. Draper Laboratory, Cambridge, Mass.

This report consists of two major parts.

Part A - Micromechanical properties of instrument grade beryllium. (description of the materials problem, instrumentation to make strain measurements in the range of 10^{-7} , and initial results.)

Part B - 10^{-8} creep measurement system (introduction, the algorithm, environmental chamber, cell proper, interferometer, summary.)



MICROMECHANICAL PROPERTIES OF INSTRUMENT GRADE BERYLLIUM

A description of the materials problem and instrumentation to make strain measurements of 10^{-7} and upwards.

Initial results.



MICROMECHANICAL PROPERTIES OF INSTRUMENT-GRADE BERYLLIUM

by

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I. Beryllium in Gyroscopes

Various grades of beryllium have found application in components of gyroscopes. Beryllium is attractive for this purpose because of its low density, high strength-weight ratio, high stiffness, and very high thermal conductivity. Gyroscopes in service require periodic realignment (sometimes as often as once a month) to correct for drift due to various causes, including dimensional instability of beryllium components. The current state-of-the-art in gyroscope design is based on a net mass shift in critical components, such as, wheels of about 10⁻⁶ inches. This mass shift is characteristic of beryllium components machined from typical commercial production lots of instrument-grade beryllium. Improved dimensional stability of the beryllium will pave the way for future advances in the design of gyroscopes. For example, mass shift stability of 10⁻⁸ inches is sought for the fourth-generation gyroscopes now being designed.

This research program has two goals. One is to develop a data base on the microcreep rates of instrument-grade beryllium for use by gyroscope designers. The other is to develop fundamental understanding of the causes of dimensional instability in instrument-grade beryllium through correlations between measurements of microplastic behavior and microstructural observations. Microplastic properties to be measured include microcreep rates, microyield stresses, and zero-load dimensional changes. Microstructural observations will be made using optical microscopy (phase-contrast mode), transmission electron microscopy, scanning electron microscopy, and electron microprobe techniques.

II. Progress to Date

This research program was initiated on October 1, 1977. A uniaxial tensile strain measuring system for reliable and accurate measurements in the range of 10^{-7} inches and upwards is now operational. Microcreep rates are being measured at stresses from 1,000 to 15,000 psi at 144 ± 0.018 °F (62.2 ± 0.01 °C) in an environmentally-stable chamber. This temperature was selected to simulate the environmental conditions in which a gyroscope's wheel operates. Temperatures of critical components of an operating gyroscope range from 125-150°F (51.7-65.5°C).

An apparatus with the environmental and electronic stability required for measurements in the 10⁻⁸ inch range has been designed and is partially-constructed. A literature review on the microplastic properties of beryllium has been completed. This review includes a survey of the microyield stresses for production lots of instrument grade beryllium manufactured during the last two years. Some of the survey results appear in Part V of this report.

III. Testing System for Measuring 10⁻⁷ Strain

Figure 1 shows a testing system capable of measuring displacements below the micro-inch range. The heart of this system is an extensometer utilizing parallel-plate capacitors, which is described below. The extensometer fastens to the cylindrical test specimen via machined ribs, Figure 2. The specimen is installed in a creep frame of conventional manufacture. Dead load is applied via a lever-and knife-edge linkage through ball-end joints on the specimen. The creep frame is mounted on a concrete pedestal to minimize building vibrations. In operation, a foam insulating cabinet surrounds the specimen and ball-end joints to provide finely-tuned environmental control. Almost all of the electronic equipment for measurement of displacement, temperature, and load is placed on a standard relay rack, as shown in Figure 1. The entire system is located in a remote basement room with good temperature and humidity control.

The extensometer has been described in detail elsewhere. 2 It consists of three independent parallel plate capacitors arrayed at 120° to one another about the specimen circumference. The output of this device is linear with displacement through use of appropriate circuitry. 3 In its present state of refinement, the extensometer has an operating resolution of about 4×10^{-8} inches.

The average displacement determined from the independent strain probes represents the displacement along the axial centerline of the test specimen. This conclusion is arrived at from geometric analysis. Consequently, all axial strains are calculated from the average displacement. Bending strains can be calculated by comparing the average strain with the strain determined at each strain probe.

Bending arises from misalignment.

Alignment is an important consideration in microcreep measurements. Good alignment is promoted by careful machining of the specimen and testing system components, and by thoughtful design of the load train. This subject has been reviewed thoroughly by Christ and Swanson. Because misalignment cannot be completely eliminated in the best of testing systems, it has been suggested that misalignment characteristic of microstrain measurements be represented by the tangent of the angle between the tensile axis and the normal to the bend plane. Typical numbers for the present equipment range between 10⁻⁵ radians (very good alignment) to 10⁻⁴ radians (moderately good alignment).

Two techniques were used to calibrate the extensometer. First, a fringe-counting laser interferometer was used as an absolute standard for measuring length changes. The calibration constant determined using this technique was 0.0352×10^{-6} inches per dial division on the decade transformer of the bridge circuit. A second technique for calibration was to calculate the displacement of an elastically-deformed beryllium sample from a handbook value of Young's modulus, 42.4×10^6 psi. The calibration constant was 0.03×10^{-6} inches per dial division. These two calibration constants agree well with one another.

The microstrain data reported in Figures 3-5 were calculated using a calibration constant of 3.5×10^{-8} inches per dial division.

Temperature control to about 0.018°F (0.01°C) is needed to make meaningful displacement measurements in the 10⁻⁷ inch range. Using the heat from light bulbs, temperature in the environmental chamber is controlled at 144°F (62.2°C) with a standard deviation of 0.018°F (±0.01°C). Successful runs have been made for an accummulated time of 1200 hours. Temperature was measured using a single thermistor mounted on the specimen just below the gage section.

IV. Initial Test Results

Initial results obtained with the testing system serve two purposes. First, they provide a demonstration of the testing system capabilities. Secondly, they provide a preview of some important features of the microcreep behavior of instrument grade beryllium.

These initial results were obtained on a single specimen of the type shown in Figure 2. Test temperature was 144 ± 0.018 °F (62.2 ± 0.01 °C). The specimen was stress relieved after machining with the following treatment*:

- o Heat slowly (at <100°F per hour) to 1450°F
- o Dwell at 1450°F for 1 hour to allow recovery
- o Cool slowly (at <100°F per hour) to room temperature

Figures 3-5 show initial microstrain results from dead-weight loading of this specimen. Several sequential loadings are indicated in the upper half of Figure 3. Once loaded, the specimen was held at stress for at least one week. Between loadings the specimen was completely unloaded. Some refinements were made to the testing system during the three loading cycles in the first 400 hours, in order to improve the quality of the data. About 5 microinches per inch (hereafter, "microstrain"

This treatment is the standard practice followed at the Charles Stark Draper Laboratory, Inc., Cambridge, Massachusetts.

will be used to signify "microinches per inch") accumulated during these three loading cycles, as shown in Figure 4. The cumulative plastic strain for all loading cycles is reported in Figure 4. Typical microcreep curves at 5,000 and 1,000 psi are shown in Figure 5.

These initial results show that the testing system operates according to design expectations. Displacement resolution is about 4×10^{-8} inches. Temperature control is held to about $0.018^{\circ}F$ ($0.01^{\circ}C$), allowing for routine displacement measurements from 1×10^{-7} inches upwards. The system has good alignment.

Some interesting aspects of the microcreep behavior of instrument grade beryllium show up in these initial results.

- o Figures 3, 5. Measurable microcreep shows up at stresses less than the nominal microyield strength (hereafter "MYS") of 8.6 Ksi reported by the manufacturer. A typical microcreep rate is 1 x 10⁻⁸ per hour, i.e., 1 micro-inch in 100 hours.
- o Figures 3, 5. Microcreep is linear with time, at least up to 140 hours.

 The expected parabolic response might show up with longer times at stress.
- o Figure 3. Microstrain hardening reduces the microcreep rate substantially.

 The amount of microstrain in a sequentially-loaded specimen does not increase in proportion to increasing stress, apparently due to microstrain exhaustion.
- o Figure 4. The cummulative plastic strain from successive loadings, including stresses above the MYS, is less than 20 micro-strain.
- V. Analysis of Manufacturers' Microyield Strength (MYS) Data

For lack of a better method, gyroscope designers estimate microcreep behavior from measurements of MYS. The (unproven) assumption is that the higher the MYS, the better the microcreep resistance should be. One of our long-term objectives is to assist designers by evaluating this assumption by looking for a correlation between MYS and microcreep resistance. As a starting point, we have solicited MYS

data from manufacturers. MYS data going back for 2 years on commercial production lots of instrument-grade beryllium have been obtained and analyzed. Results are shown in Figure 6. The following observations can be made:

- Figure 6a Manufacturer A
 - o Over the two-year time interval, MYS ranged from 8,000-14,000 psi
 - o MYS and scatter-band width tended to increase in the more recent production lots.
 - o Within a given lot, there is a noticible variation of MYS from one location to another within a pressing. Statistical analysis is being carried out to formally evaluate this point.
 - o There appears to be an increase in the MYS by applying stress relief and thermal cycling treatments.
- Figure 6b Manufacturer B
 - o Over the two-year time interval, MYS ranged between 8,000 and 14,500 psi.
 - o MYS and scatter-band width tended to increase in the more recent production lots.
 - o Within a given lot, there is a noticible variation of MYS from one location to another within a pressing. Statistical analysis is being carried out to formally evaluate this point.

VI. Future Work

Eight test specimens of the type shown in Figure 2 are currently being machined at the C.S. Draper Laboratory, Cambridge, Massachusetts. The machine shop there has special facilities for handling toxic beryllium particles formed during machining. Once in hand, these specimens will be tested in the system described in Part III of this report. Testing will be carried out over a range of stresses (1/2 MYS to 1 1/2 MYS) and temperatures (68 to 180°F). The microcreep curves developed will be of use to designers working with instrument grade beryllium. Hopefully, a Zener-Hollomon plot can be developed to assist designers.

Micromechanisms of deformation will be looked into using transmission electron microscopy and other techniques to probe the microstructure. The dislocation content and configuration of undeformed and deformed specimens will be examined and compared. An attempt will be made to identify the dislocation sources and barriers. It is expected that these results will serve as a basis for future development of grades of beryllium exhibiting good dimensional stability.

The following areas are suggested as supplements to the original program if support can be found.

- Establish several testing systems of the type described in Part III.
 Multiple testing systems would increase the data rate substantially.
- 2. Measure microyield strength and microcreep resistance of several grades of beryllium, to establish whether or not a correlation exists between these two micromechanical properties.
- 3. Determine how various thermal cycling treatments affect micromechanical properties and deformation mechanisms of instrument grade beryllium.

VII. Acknowledgements

Mr. John McCarthy, C.S. Draper Laboratory, Inc., Cambridge, Mass. provided helpful background information about beryllium in gyroscopes and has guided the machining of damage free test specimens at the C.S. Draper Lab for the N.B.S. testing program. Gyroscope design information has been provided by Mr. Fred Petrie, C.S. Draper Laboratory, Inc., Cambridge, Massachusetts and Mr. Fred Hallock, Northrup Precision, Waltham, Massachusetts. Useful discussions about micromechanical testing have been held with Mr. C. W. Marshall, Battelle-Columbus, Ohio and Mr. A. Gross, Autonetics - Rockwell International Corporation, Anaheim, CA.

Mr. Roger Paquin, Perkin-Elmer, Stamford, Conn. has contributed interesting discussions about precision applications of beryllium mirrors for optical equipment. Mr. Fullerton-Batten of Kawecki-Berylco, Hazleton, Pennsylvania provided some of the data appearing in Part V. Mr. William Becker and Mr. J. Stonehouse, Brush-Wellman, Inc., also provided some of the data in Part V.

Figure Captions

- 1. View of testing system used for microcreep measurements.
- 2. Microcreep test specimen for measurement in the 10^{-7} to 10^{-6} inch range.
- 3. Loading sequence and strain-time history for specimen Be-1.
- 4. Cummulative strain from sequential loadings of specimen Be-1.
- 5. Microcreep behavior of specimen Be-1 at 5,000 and 1,000 psi.
- 6a. MYS versus batch number from Manufacturer A.
- 6b. MYS versus batch number from Manufacturer B.



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 Belfour Stolen Inc.



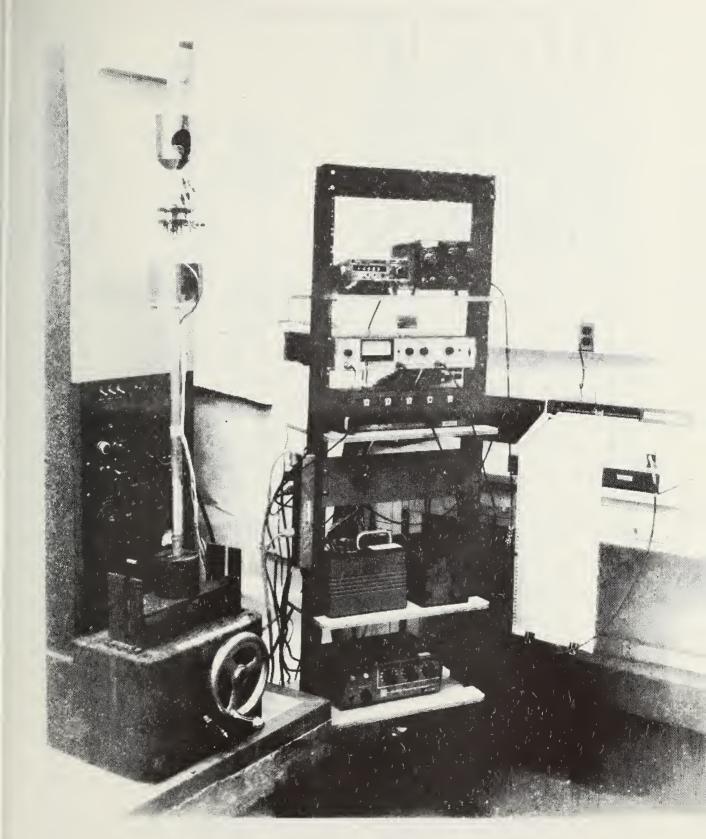
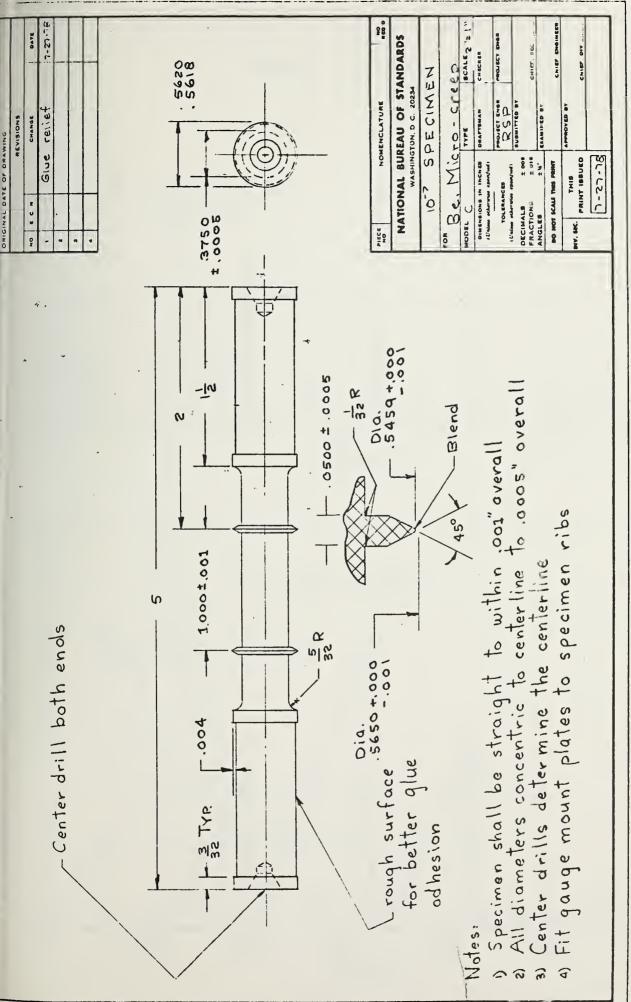


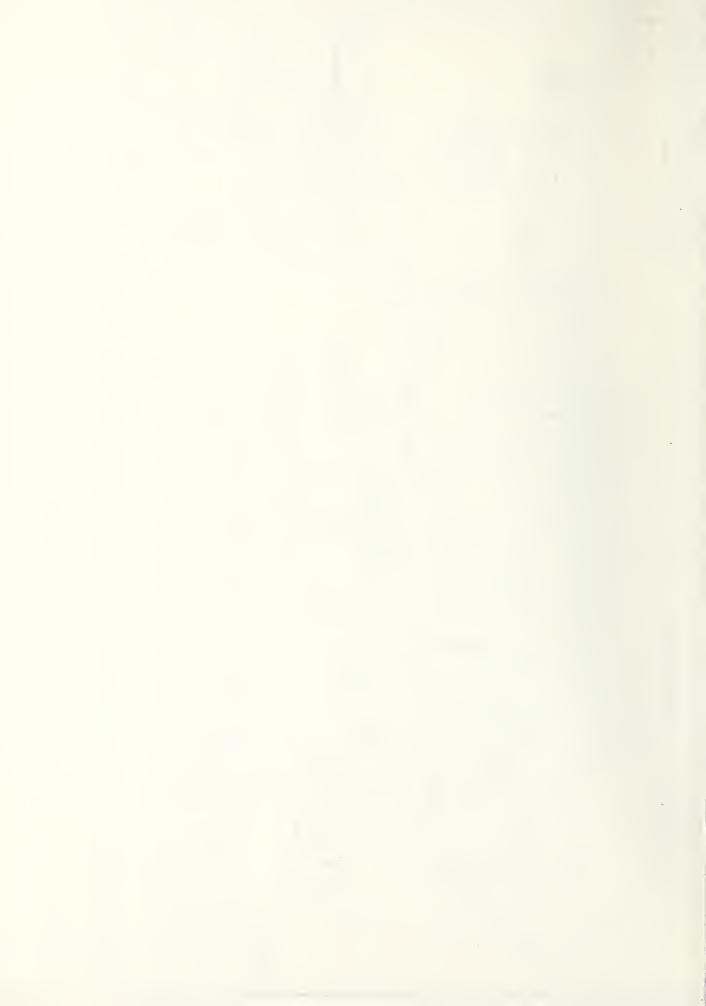
Figure 1

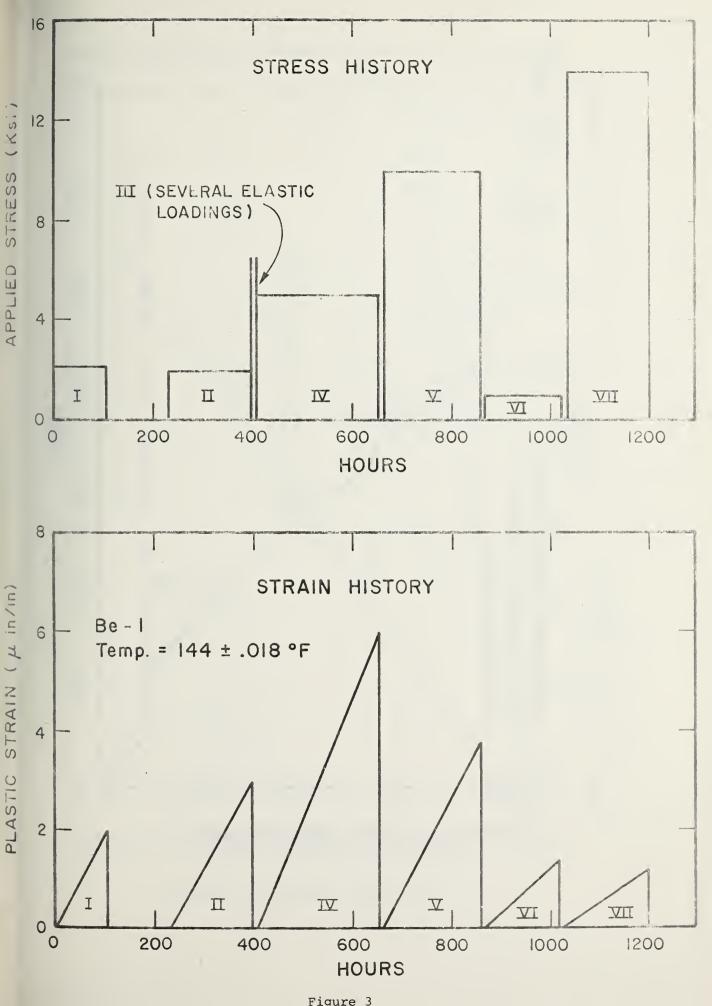


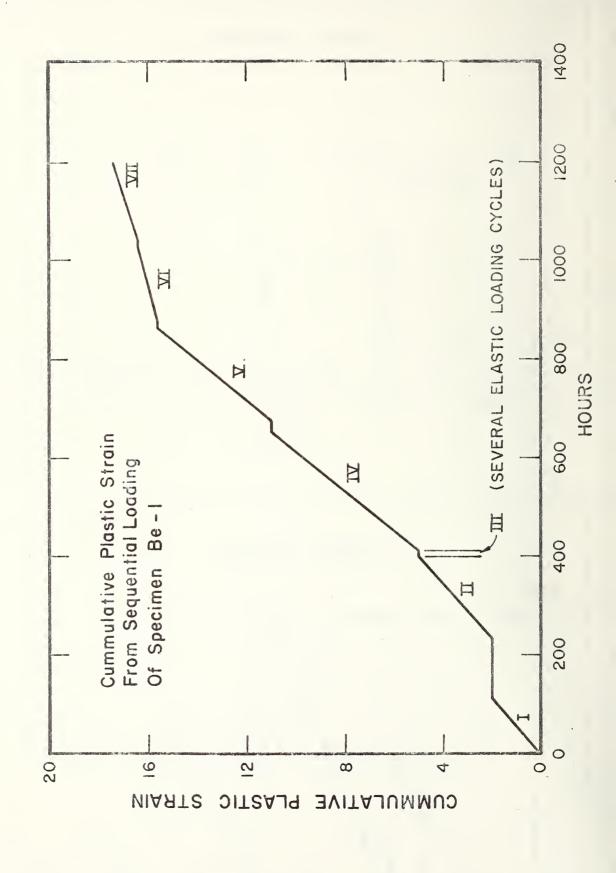


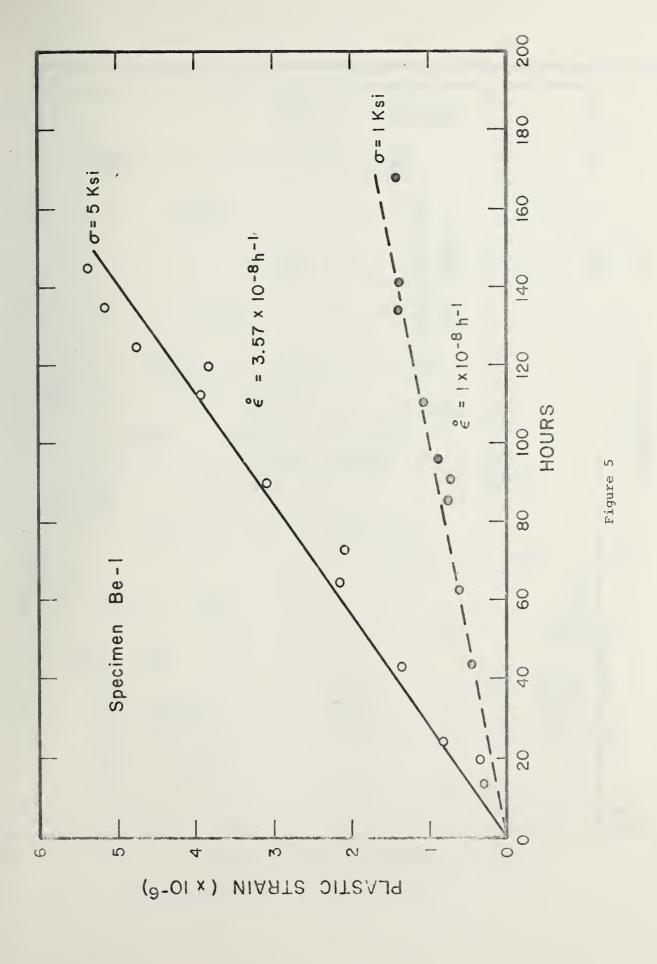
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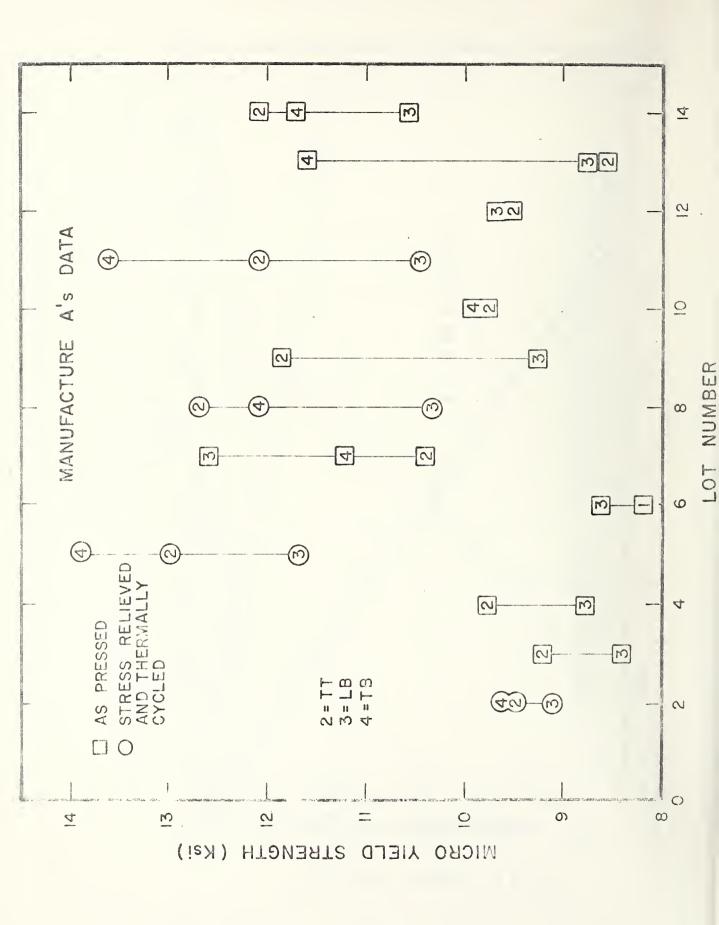
Figure 2

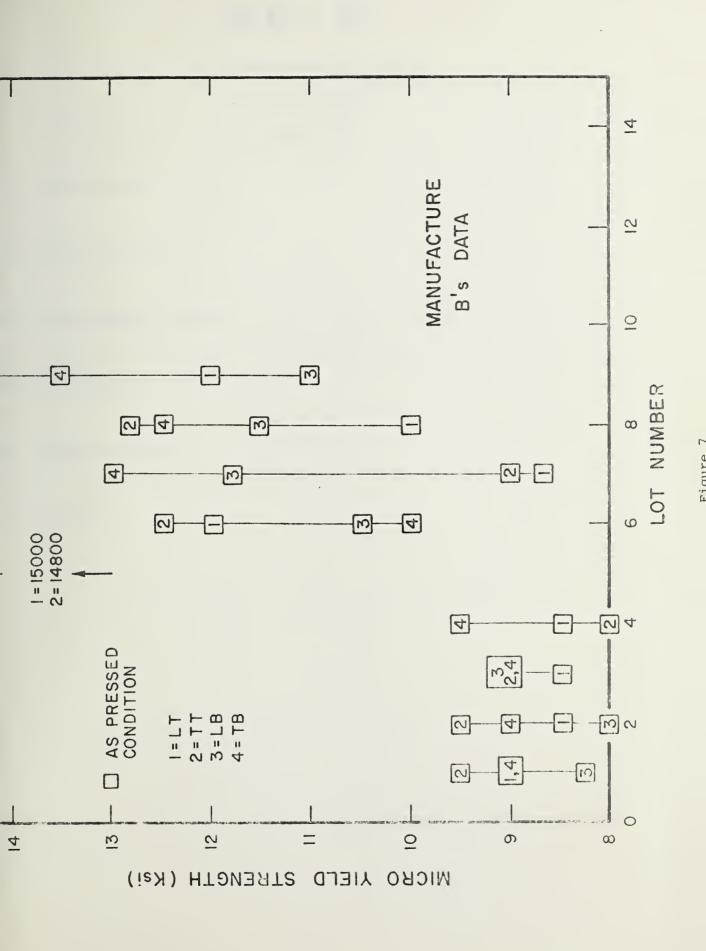














PART B

10⁻⁸ Creep Measurement System

Progress Report

August 1978

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II The Algorithm

III Environmental Chamber

IV Cell Proper

V Interferometer

VI Summary



10⁻⁸ Creep System

1. INTRODUCTION

The goal of this portion of the project was to design, build, and test a system for the measurement of microcreep in Beryllium test specimens. It was decided early in the project that the specimens were to be tested around 45 °C, at loads up to 10,000 psi in tension. The time duration of the tests was set at a minimum of three (3) months and the accuracy at 10⁻⁸ inches. These conditions lead to certain measurement constraints that are summarized in Table 1. On the basis of these constraints several design decisions were made. They are:

- a) Laser interferometry was chosen as the length measurement sensor.

 No other system known to us has the requisite stability over the required time.
- b) A modified atmosphere was chosen as the most cost effective way to reduce the effect of refractive index changes on the interferometric length scale.
- c) A two stage conduction thermostat was chosen to obtain the required thermal stability.
- d) Dead weight loading was chosen as no other loading system known to us provides the required stability.

e) A measurement algorithm was chosen to provide a consistent cross check and give assurance that the apparatus was meeting design specifications.

As of the time of this report the design is completed and construction well advanced. In the next 4 sections of this report we will describe the algorithm, the enclosure design, the sample containers, and the interferometer.

II. The Algorithm

In any measurement system the measurement algorithm is predicated by the most stringent restriction to be placed on the measurement to be made. In this instance, two restrictions combined to provide a very difficult measurement problem. Specifically, the combination of high accuracy (10^{-8} inches) and long time (>3 months) dominates all other considerations.

In any algorithm two conditions must be met. First the measurements should provide data of accuracy sufficient for the intended use and secondly, there must be enough cross checking to insure that the measurement system is functioning to the required degree of accuracy. In order that these conditions be met, in spite of the two restrictions previously mentioned, we have designed the experiment around a 6/2 time corrected measurement sequence. In such a sequence there is one "master", one "check standard", and four "unknowns".

Ideally such a system should include temperature checking, instrument checking and sample checking. To meet these objectives we have decided upon the following samples:

- 1) A cervit block, nominally of 4 inches in length which has an effective expansion coefficient of 0.034 ppm/°C.
- 2) A well aged gage block, again of 4 inches in length, which has a coefficient of 11.5 ppm/°C, quite close to that of Beryllium.

The combination of samples 1 and 2 is intended to act as a "master" and a "check standard". Since both materials are of high stability (1) we can expect to get from 1, a check on the interferometry, and from the differences between 1 and 2, a check on the temperature stability of the system.

All the other samples are Beryllium. They are:

- 3) An unloaded Be sample from the same batch as the samples under load and
- (4,5 Be samples under loads of nominally 10,000 psi, 5000 psi and 2500 psi.

Sample 3 serves to access the long term dimensional stability of

Be, which is, to our knowledge, unknown. The other three samples are to

provide the necessary data to obtain the creep curve at a single temperature.

Table 2 shows the planned sample identification and the measurement sequence for each time a measurement is made. The full details of the algorithm design and the computer program to be used for data reduction have been explained by Reeve. (2) The principle, briefly outlined, is to establish the process standard deviation by the repeated measurements, to check for process control by testing the differences between the master and check standard and to remove any operator bias by using pseudo random sequencing.

III. The Environmental Enclosure

In order to implement the algorithm described in Section II, It was necessary to design an enclosure that would contain 6 samples in a controlled temperature and controlled atmosphere environment. Futhermore, such an enclosure had to allow for dead weight loading and maintain the samples precisely aligned for optical gaging.

Figures 1, 2, and 3 show the enclosure layout. The device consists of a rigid base plate to provide structural strength, a two stage

conduction thermostat built around this base plate, and provision for loading three of the six samples it can contain.

Two features of the design are of interest. First, its successful operation requires five (5) thermal control systems (heater, sensor, bridge and feedback loop). Two of these systems control the main cylindrical shells. The outer shell is to be controlled to ±.01 degrees at a temperature slightly below the nominal 45 °C. The inner shell is then controlled only slightly above the outer shell to insure isothermal conditions. Thermostats of this type have developed stabilities of ±10 µK over many months. (3) The other three loops are to be used for canceling the heat leaks necessitated by the load bearing rods that emerge from the thermostat to the load frame. Matched thermistor pairs will be used, one in the inner case and one on the rod, to sense and correct for any temperature differences that arise. Figures 4 and 5 show the bridge circuit and feedback loop circuits to be used for these control operations.

The other feature was necessitated by the requirement of atmospheric control coupled with deadweight loading. In order to seal the device yet still allow nearly frictionless motion of the load rods, new sealing techniques had to be tried. At the stage where the rod passes through the base plate, ferrofluidic seals have been incorporated to provide this seal.

At the time of this writing, the enclosure construction is nearing completion and we expect to do the initial assembly before October 1, 1978.

IV. The Sample Cell

Because of the modular nature of the enclosure design each of the samples required a special cell which is an integral part of the loading mechanism. Figure 6 shows the layout of one of these sample containers. It is designed to have two adjustments of sample angle with respect to the loading mechanism. One of these is furnished through the enclosure inner shell base plate. This adjusts the angle of the bottom grip, which is constrained by a high quality linear bearing, with respect to the loading member. The other adjustment alters the angle of the top grip with respect to the bottom. This adjustment has a coarse control, screws, which are used in initial alignment and a fine adjustment provided by piezo stacks which will be electrically controlled in the measurement process. Sample alignment to within +1 second of arc is expected.

V . The Interferometer

Initially it was thought by the authors that the interferometry could be done with a simple Michelson configuration. This proved impossible

in prototype form due to errors caused by our inability to align samples to the degree of angular reproducibility (~ 0.01 arc sec) required to obtain 10^{-8} accuracy with this type of interferometer.

A second interferometer with the requisite insensitivity to angle was then built (4). It is diagrammed schematically in Figures 7 and 8.

Figure 7 shows it in a form that allows ready understanding of the principle involved and Figure 8 the configuration as it will actually be used.

Briefly the device works as follows: a two frequency beam of polarization encoded laser light is incident upon the beam splitter. One frequency f 2 is reflected off the beam splitter, into a corner cube, reflected from the beam splitter again and returned to the detector (at the same angle as the incident angle). The other beam f_1 passes through the beam splitter and quarter wave plate and is reflected off the sample. It returns through the quarter wave plate to the beam splitter but now, because of the double passage through the quarter wave plate, is reflected at the beam splitter interface and into the other cube corner. The cube corner retroreflects it back to the plane reflector, preserving the angle, so that it strikes the plane mirror at the same angle as it emerged after the first reflection. It therefore comes off the mirror after this second reflection at its original incident angle and returns towards the beam splitter. Double passage through the quarter wave plate again has rotated the polarization 90°, however, and the beam now passes out of the interferometer. The double reflection and retroreflector thus make this device highly insensitive to angle (up to several degrees). Angular information

may easily be extracted, however, by a 45° rotation of the quarter wave plate. This feature allows autocollimation of the reflection for sample alignment within the +1 sec specified previously.

A prototype system like that shown in Figure 8 has been built and tested and the final interferometer designed and built.

The electronics to be used for the phase detection of the heterodyne beat notes between the two optical beams is shown in Figure 9. The detector and most electronics (amps, phase meter, reference oscillator, H. P. counter) have now been ordered but procurement delays have been encountered. We expect initial testing of the final system to begin sometime in November.

VI. Summary

We now believe that all the serious design problems for this system have been overcome and expect to begin debugging of the final setup on or about October 1. Data acquisition should commence three months from that date. Additional expenditures of about \$10-20 K in equipment are anticipated, mostly for a dedicated laser and counter system.

Table 1	_	Measurement	Constraints	Š

	Precision or		•	
Parameter	Stability	Accuracy	Reason	
Length	$\pm 10^{-8}$ inches	$\pm 10^{-8}$ inches	Design Specs	
Time	>10 ⁶ sec	NA	Design Specs	
Predicated by 1 and 2				
Temperature	<u>+</u> .001 °C	<u>+</u> .1 °C	Thermal Expansion	
Load	<u>+</u> 10 ⁻⁵	<u>+</u> 1%	Elastic Deformation	
Atmosphere	<u>+</u> 0.01 mm (air)	<u>+</u> 0.01 mm	Length Scale Stability	

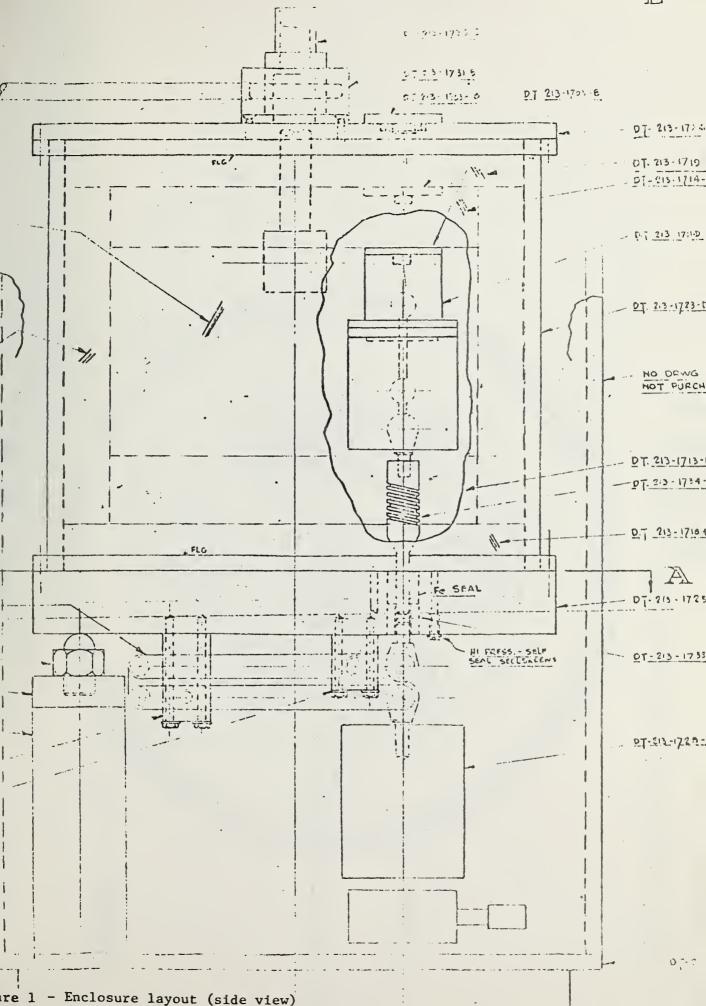
Table 2 - Algorithm Design

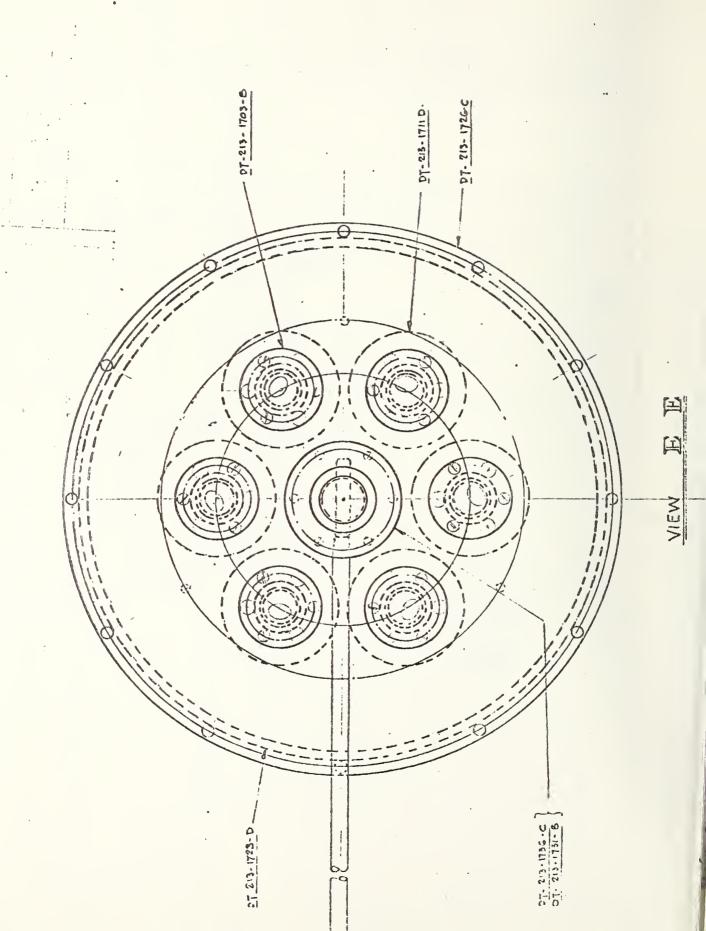
A) Sample Specification

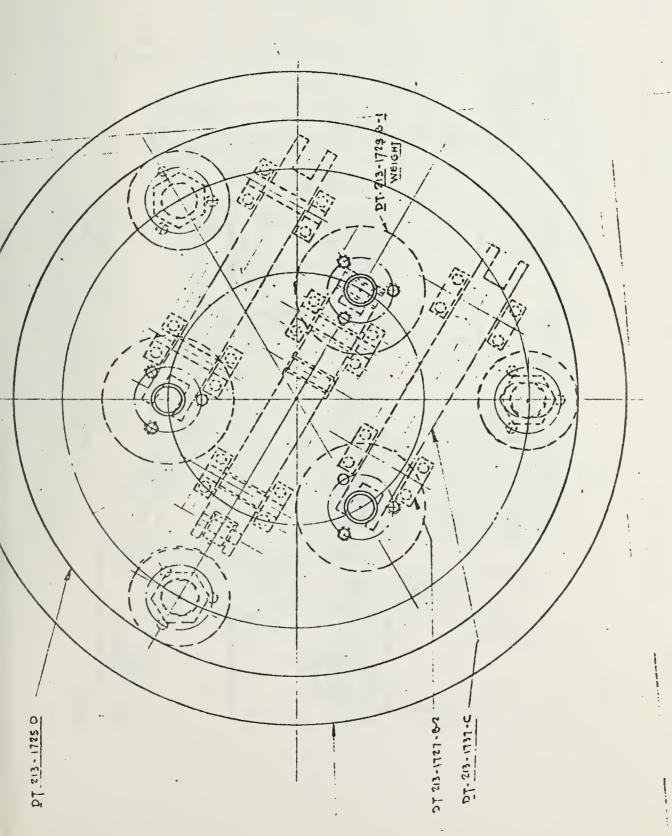
Number	Identification
1	Cervit
. 2	Steel
3	Be, o=10,000 psi
L,	Be,σ=5,000 psi
5	Be, σ=0
6	Be,σ=2,500 psi

B) Measurement Sequence - Single Measurement

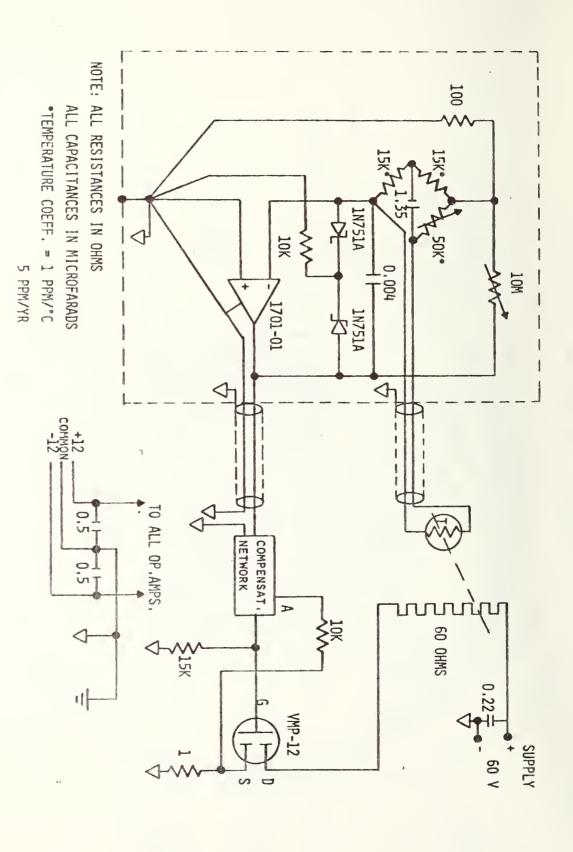
Block #	Order	
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2	3,4,3,1,3,5,3	
3	4,5,4,1,4,6,4	
4	5,6,5,1,5,2,5	
5	6,2,6,1,6,3,6	







.Figure 3 - Enclosure layout showing loading system (bottom view)



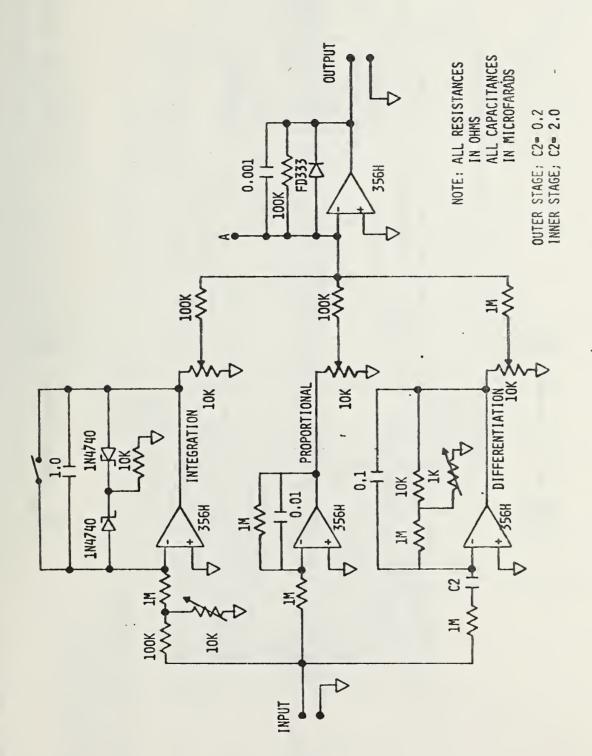


Figure 5 - Feedback Loop

Figure 6 - Sample cell layout

SECTION K

INTERFEROMETER ELECTRONICS Fa F. + 0 DETECTOR LASER IN DUAL H.P. COUNTER SCHMIDT MODIFIED TRIGGERS F REFERENCE OSCILLATOR VOLTAGE CONTROLLED MIXR MIX ERROR 5-F Fp+ Ø Fo FREQUENCY COMPARATOR AND FEEDBACK SIGNAL CONDITIONING F LOCAL PHASE METER OSCILLATOR

PHASE (FRINGE

FRACTION)

FRINGE COUNT

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ston, Massachusetts 02210 htract Moniter: Dr. Frank Gardner			14. Sponsoring Agency Code
PPLEMENTARY NOTES			

ISTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bliography or literature survey, mention it here.)

This research program was initiated on October 1, 1977. It is sponsored by e Office of Naval Research. The objective of the program is to evaluate and derstand the micromechanical properties of beryllium and other instrument terials for use in gyroscopes, so that dimensional stability can be improved. proved dimensional stability is expected to lessen the need to periodically ign gyroscopes in service.

1) RORDS (six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper unc; separated by semicolons)

ryllium; dimensional stability; gyroscope; instrument materials; microcreep; cromechanical properties.

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